LANNATE (METHOMYL) RESIDUE ON YOUNG HEAD LETTUCE IN IMPERIAL COUNTY, CALIFORINA - JANUARY 1976

Ву

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#### Introduction

Lannate (methomyl) is a carbamate insecticide-nematicide. This pesticide provides broad-spectrum control of insects on a variety of vegetables, field crops, some fruit crops and ornamentals. The major uses of this pesticide in California are on head lettuce, tomatoes, and sugar beets. There were 206,056 pounds of methomyl applied to over 348,000 acres of head lettuce in California for 1975. This pesticide is formulated in a water-soluble 90 percent powder as well as in a liquid concentrate containing 1.8 pounds/gallon.

The acute oral  $LD_{50}$  in male rats has been shown to be 17 mg/kg. Acute dermal  $LD_{50}$  of a 52.8 percent water slurry for rabbits has been shown to be greater than 5,000 mg/kg. The toxic effects of methomy1 are related to blood and tissue cholinesterase inhibition.

The preharvest interval for Lannate application to head lettuce as stated on the label is seven days when applied at a rate of 1/4 to 1/2 pound of methomyl/acre and 10 days when applied at a rate greater than 1/2 pound/acre. No more than 1 pound/acre may be applied to head lettuce. The residue tolerance for methomyl on leafy vegetables (except cabbage) is 0.2 ppm.

#### Application and Sampling

In order to measure surface, penetrated and total residues from Lannate application to young head lettuce, a field in Imperial County, California, was sampled immediately after application of Lannate and then at approximately 24-hour intervals for four days. Thimet and Thurcide were also applied to the field at the same time.

Date Application Rate Per Acre

January 16, 1976

Lannate - 3 pts.
Thimet 600-1.33 pts.
Thurcide - 6 lbs. in 7 gal. water

Three samples, each consisting of about 100 leaf discs, 2.5 cm in diameter, were collected from the leaves of young lettuce plants. The minimum and maximum daily temperatures for El Centro in Imperial County, California, are given in Table I; they averaged 43.0 and 80.3 degrees F., respectively during the study. There was no rainfall during the study period.

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#### ANALYTICAL PROCEDURES

The procedure used for the extraction of dislodgeable, penetrated, and total residues from leaf punches was originally published by Gunther in "The Bulletin of Environmental Contamination and Toxicology," 9 243-249, 1973.

#### EXTRACTION:

The sample container and leaf punches are weighed and the gross weight recorded.

#### DISLODGEABLE RESIDUE

- 1. Fifty mls of water and approximately 4 drops of Sur-Ten solution (1:50) is added to the sample containers. The containers are capped and placed in a multi-purpose rotator and rotated at 30 cycles/min. for 60 min. The aqueous solution is decanted through a glass wool plug into a 500 ml separatory funnel.
- 2. The punches are rotated a second time, using 50 mls of water and 4 drops of Sur-Ten solution, for 30 min. This is added to the first extraction.
- 3. The sample is then hand shaken for approximately 10 secs with 30 mls of water. The container is drained into the separatory funnel with the first two extractions.
- 4. The aqueous solution is extracted three times with 50 ml of ethyl acetate. The ethyl acetate is filtered through sodium sulfate into a glass stoppered mixing cylinder and the volume is recorded. The solvent is mixed in the cylinder. An aliquot is decanted into a teflon capped bottle and stored in a freezer prior to cleanup and analysis.

#### PENETRATED RESIDUE

- 1. After the last water rinse is drained for the dislodgeable residue, the punches are transferred to a blender jar. The empty sample container is weighed and the net weight of the punches recorded.
- 2. Approximately 50 gms of sodium sulfate and 100 mls of ethyl acetate are added.
- 3. The sample is blended at high speed for 3 minutes, keeping the blender cup cool by immersing it in a container of cool water. The blender cup is removed and the sample allowed to settle.
- 4. An aliquot is decanted into a teflon capped bottle and stored in a freezer prior to cleanup and analysis.

#### TOTAL RESIDUE

- 1. The leaf punches are transferred to a blending jar. The empty sample container is again weighed and the net weight of the punches recorded.
- 2. Approximately 50 gms of sodium sulfate and 100 mls of ethyl acetate are added.
- 3. The sample is blended at high speed for 3 minutes, keeping the blender cup cool by immersing it in a container of cool water. The blender cup is removed and the sample allowed to settle.
- 4. An aliquot is decanted into a reflon capped bottle and stored in a freezer prior to cleanup and analysis.

#### CLEANUP

Transfer sample to a 250 ml separatory funnel. Rinse the flask with 50 ml  $0.01~\mathrm{N}~\mathrm{H}_2\mathrm{SO}_4$  and add to separatory funnel. Rinse the flask with 50 ml water and add to funnel. Agitate gently for one minute. Vigorous agitation will cause emulsions. Insufficient agitation will result in low recoveries. After phase separation, discard the ethyl acetate. Repeat the ethyl acetate partitioning if necessary. Multiple extractions will not depress recoveries. Extract the aqueous layer 3 x 50 with  $\mathrm{CH}_2\mathrm{Cl}_2$ , drying each extract with sodium sulfate.

#### ANALYSIS

For liquid-chromatographic determination, concentrate to an appropriate volume and inject.

#### LC Conditions

Chromatronix 3500, Schoeffel detector at 233 nm Partisil 10 micron column 25 cm long 6% MeOH in ethyl ether at 2 ml/min

#### Retention times

Lannate

5 min

100 ng gives about 50% deflection at 0.01 AUFS

#### Results

The dislodgeable, penetrated and total residue of methomyl found are given in Table II. Both dislodgeable and penetrated residues declined to less than 10 percent of their initial values in four days (Graph I). Surface and penetrated residues declined at approximately the same rate. The decline of residues in and on very young lettuce plants resulted from both dilution by rapid plant growth and from chemical degradation and dissipation.

Table I

Maximum and Minimum Air Temperatures in El Centro, California

January 14-20, 1976

January 1976

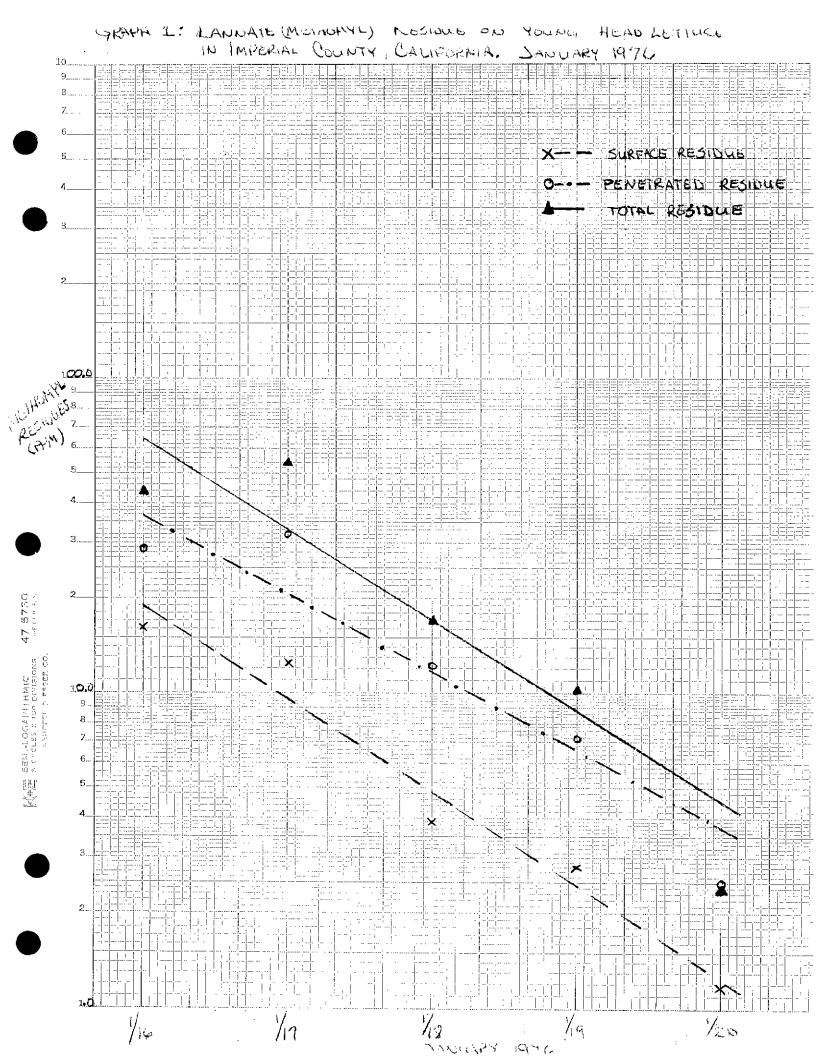
#### Temperatures

<u>Dates</u>		Maximum	Minimum
14		74°	35°
15		80°	34°
16		83°	42°
17		80°	43°
18		78 <b>°</b>	45°
19		78°	40°
	Average	80.3°	43.0°

Table II
Residues of Methomyl (in PPM) Following Application
of Lannate to Young Head Lettuce in Imperial
County, California in January 1976

Date		<u>Sample</u>	Surface	<u>Penetrate</u> d	<u>Tota</u> l*
January	16	1	17.4	34.5	;
January	16	2	15.2	23.4	
January	16	3			44.6
January	17	4	10.4	29.9	
January	17	5	14.6	34.2	
January	17	6			54.0
January	18	7	4.2	12.3	
January	18	8	3.6	12.4	
January		9			17.1
January	19	10	3.1	9.1	
January		11	2.55	5.15	
January		12			10.3
January	20	13	1.16	2.52	
January		14	1.22	2.49	
January		15		20-17	2.43

<sup>\*</sup> The total column represents separate analyses of separate jars of leaf discs presumably similar to the samples used to determine the surface and penetrated samples.



## Addendum to HS-312 Reaclculation of Dislodgeable Residues

# Results of Analysis of Lettuce Leaves for Dislodgeable Residues of Methomyl

Sample Interval	Residue (ug/cm2)		
=======================================			
1 hr.	.352	.388	
l day	.221	.262	
2 days	.088	.073	
3 days	.063	.064	
4 days	.030	-027	